## **Appendix D**

# ESEM and SEM/EDS Data for Test #3, Day-30 Corrosion Products

## **Figures**

Figure D-1.	SEM image magnified 200 times for a Test #3, Day-30 powder on the
	submerged rack. (T3~RackPowder013)
Figure D-2.	EDS counting spectrum for the powder on the submerged rack shown
	in Figure D-1. (T3RackPowder09)
Figure D-3.	SEM image magnified 500 times for a Test #3, Day-30 powder on a
	submerged galvanized steel coupon. (T3~Gal~Steel~Powder014) D-
Figure D-4.	EDS counting spectrum for the white powder on galvanized steel
	shown in Figure D-3. (CorPdct~GalStel10)
Figure D-5.	EDS counting spectrum for the dark powder on galvanized steel
	shown in Figure D-3. (CorPrdct~GalSteel11)
Figure D-6.	SEM image magnified 500 times for a Test #3, Day-30 powder on a
	submerged copper coupon. (T3~Copper~Powder015) D-1
Figure D-7.	EDS counting spectrum for the powder on copper shown in Figure
	D-6. (CorPrdct~cu12)
Figure D-8.	SEM image magnified 100 times for Test #3, Day-30 corrosion
	products on a submerged aluminum coupon.
	(T3D30CorrPrdctSubmAl039)D-1
Figure D-9.	Backscattered SEM image magnified 100 times for Test #3, Day-30
	corrosion products on a submerged aluminum coupon.
	(T3D30CorrPrdctSubmAl040)D-1
Figure D-10.	EDS counting spectrum for the corrosion products (particles) shown
	in Figure D-9. (T3D30CorrPrdtAl22)

Figure D-11.	SEM image magnified 1000 times for Test #3, Day-30 corrosion	
	products on a submerged aluminum coupon.	
	(T3D30CorrPrdctSubmAl041)D-	-16
Figure D-12.	ESEM image magnified 1000 times for Test #3, Day-30 corrosion	
	products on a submerged concrete coupon. (T3cont28)	-16
Figure D-13.	ESEM image magnified 100 times for Test #3, Day-30 corrosion	
	products on a submerged concrete coupon. (t3cont29)	-17
Figure D-14.	EDS counting spectrum for the corrosion products (particles) shown	
	in Figure D-13. (t3cont30)	-17
	Tables	
Table D-1.	Chemical Compositions for T3RackPowder09, Figure D-2	<b>)</b> -7
Table D-2.	Chemical Compositions for CorPrdct~GalSteel11, Figure D-5D-	-10
Table D-3.	Chemical Compositions for CorPrdct~cu12, Figure D-7D-	-12
Table D-4.	Chemical Compositions for T3D30CorrPrdtAl22, Figure D-10	-15

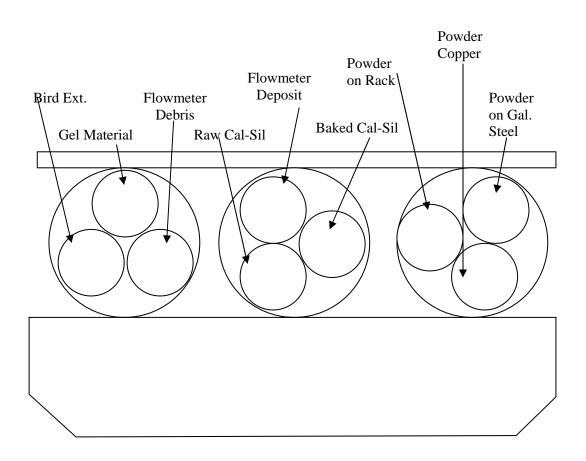
For ICET tests, one process of interest is the corrosion effect of metal and concrete coupons. One means of understanding the corrosion process is through direct examination of the corrosion products after the test is completed. For this purpose, corrosion products were collected when Test #3 was shut down (May 5, 2005). These corrosion products included (1) fine powders on a vertical piece of the submerged CPVC rack, (2) corrosion products on a submerged galvanized steel coupon, (3) corrosion products on a submerged copper coupon, (4) corrosion products on a submerged aluminum coupon, and (5) corrosion products on a submerged concrete coupon.

Corrosion products were collected by directly adhering the sample onto double-sided carbon tape suitable for SEM/EDS examination. After the samples were dried in air, an Au/Pd coating was applied to enhance the surface conductivity of the samples and to prevent possible charging problems during the SEM examination process. Based on EDS results, a semi-quantitative elemental analysis was performed after calibration of the x-ray signal using an internal standard of the microscopy. This appendix presents the SEM/EDS data that were generated on May 17, 2005. Available logbook entries for this laboratory session are included in this appendix as transcribed notes.

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### **Transcribed Laboratory Log**

# <u>Laboratory session from May 9, 2005.</u> Test #3, Day-30 Powder Deposits



#### **Powder on Rack**

T3~RackPowder013 Figure D-1 Image:  $200 \times$ 

T3RackPowder09 Figure D-2 EDS: Powder on image 013

#### **Powder on Galvanized Steel**

Image: T3~Gal~Steel~Powder014  $500 \times$  Figure D-3

EDS: CorPdct~GalStel10 White powder on image Figure D-4

014

CorPrdct~GalSteel11 Dark powder on image 014 Figure D-5

#### **Powder on Copper**

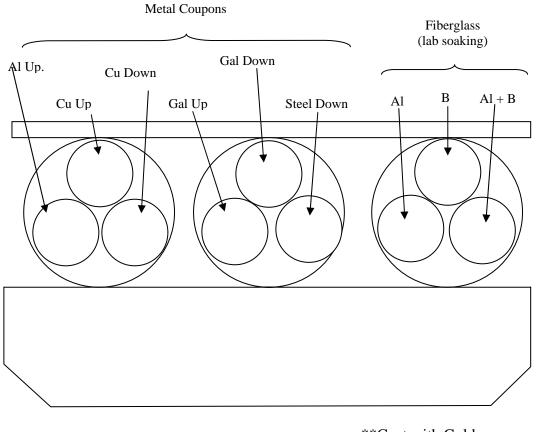
Image: T3~Copper~Powder015 500 × Figure D-6

EDS: CorPrdct\_cu12 Powder on image 015 Figure D-7

#### **Transcribed Laboratory Log**

#### Laboratory session from May 17, 2005.

Test #3, Day-30 Metal Coupons



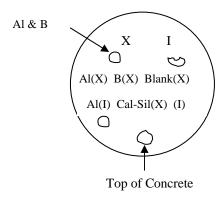
\*\*Coat with Gold

#### **Corrosion Product on Submerged Aluminum**

Image:	T3D30CorrPrdctSubmAl039	100 ×		Figure D-8
	T3D30CorrPrdctSubmAl040	100 ×	Backscattering	Figure D-9
EDS:	T3D30CorrPrdtAl22		Particles on image 040	Figure D-10
Image:	T3D30CorrPrdctSubmAl041	1000 ×		Figure D-11

#### **Transcribed Laboratory Log**

<u>Laboratory session from May 10, 2005.</u> Test #3, Day-30 ESEM.



### **Deposits on the Top of Concrete**

Image:	T3Cont28	1000 ×	Figure D-12
	t3cont29	100 ×	Figure D-13
EDS:	t3cont30	Particles on image 29	Figure D-14

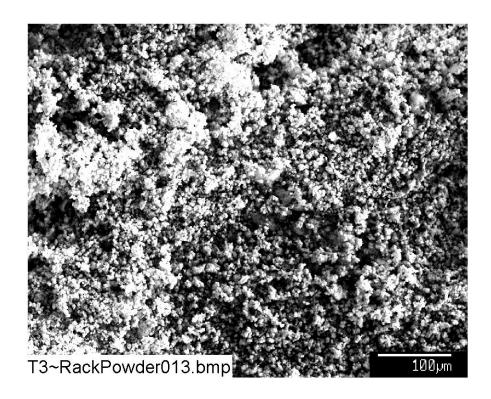


Figure D-1. SEM image magnified 200 times for a Test #3, Day-30 powder on the submerged rack. (T3~RackPowder013)

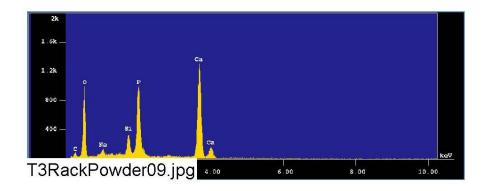


Figure D-2. EDS counting spectrum for the powder on the submerged rack shown in Figure D-1. (T3RackPowder09)

The results from the chemical composition analysis for T3RackPowder09 are given in Table D-1.

Chemical Compositions for T3RackPowder09, Figure D-2

#### 2005 May 9

Group : NRC

Sample : T3D30 ID# : 9

Comment : Powder on submerged Rack

Condition: Full Scale: 20KeV(10eV/ch,2Kch)
Live Time: 60.000 sec Aperture #: 1
Acc. Volt: 15.0 KV Probe Current: 1.608E-09 A
Stage Point: X=23.811 Y=58.398 Z= 9.938

Acq. Date : Mon May 9 15:02:46 2005

Eler	ment	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Back	ground
0	K	Normal	0.25- 0.7	7 16.4417	0.0037	6188	7 66
Na	K	Normal	0.81- 1.2	7 0.4147	0.0011	448	/ 64
Si	K	Normal	1.50- 2.0	1.8593	0.0006	2708	/ 288
P	K	Normal	1.75- 2.3	3 10.0274	0.0058	9012	/ 160
Ca	K	Normal	3.39- 4.3	22.3191	0.0043	15798	/ 22
C	K	Normal	0.09- 0.40	0.0345	0.0005	19	/ 166

Chi square = 55.4587

Element	Mass*	Atomic%	ZAF	Z	A	F
0	48.147	67.3988	1.8951	0.9734	1.9469	1.0000
Na	0.977	0.9516	1.5243	1.0269	1.4843	1.0000
Si	3.302	2.6332	1.1493	0.9767	1.1837	0.9941
P	13.197	9.5423	0.8517	1.1722	0.7283	0.9977
Ca	34.174	19.0962	0.9909	0.9956	0.9953	1.0000
C	0.203	0.3778	3.7963	1.0208	3.7192	0.9999

------

Total 100.000 100.0000

Normalization factor = 1.5452

Total 100.000 100.0000

Normalization factor = 2.1120

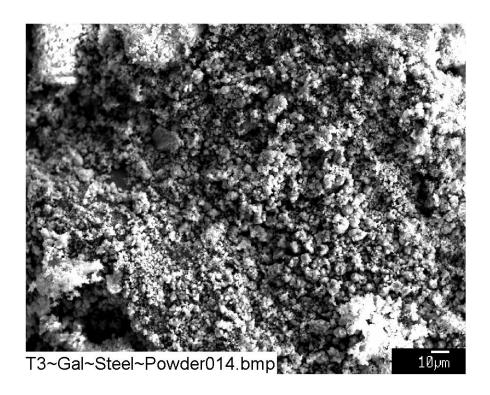


Figure D-3. SEM image magnified 500 times for a Test #3, Day-30 powder on a submerged galvanized steel coupon. (T3~Gal~Steel~Powder014)

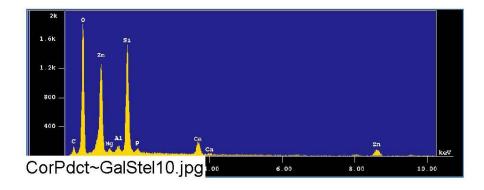


Figure D-4. EDS counting spectrum for the white powder on galvanized steel shown in Figure D-3. (CorPdct~GalStel10)

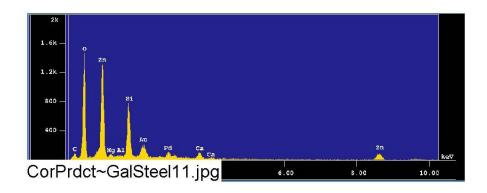


Figure D-5. EDS counting spectrum for the dark powder on galvanized steel shown in Figure D-3. (CorPrdct~GalSteel11)

The results from the chemical composition analysis for CorPrdct~GalSteel11 are given in Table D-2.

Chemical Compositions for CorPrdct~GalSteel11, Figure D-5

#### May 9 2005

: NRC Group

: T3D30 ID# : 11 Sample

Comment : Corrosion product of Gal-Steel

Acq. Date : Mon May 9 15:23:47 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Back	ground
ОК	Normal	0.25- 0.77	24.6017	0.0044	9259	/ 71
Mg K	Normal	0.97- 1.57	0.1342	0.0003	211	/ 424
Si K	Normal	1.50- 2.05	4.3383	0.0008	6318	/ 94
Ca K	Normal	3.39- 4.30	1.4823	0.0016	1049	/ 28
Zn K	Normal	8.22-10.03	13.8053	0.0096	1501	/ 6
Al K	Normal	1.19- 1.83	0.1257	0.0003	196	/ 77
CK	Normal	0.09- 0.46	0.0000	0.0000	0	/ 254

Chi square = 39.1864

Element	Mass*	Atomic%	ZAF	$\mathbf{z}$	A	F	
0	47.383	72.4077	0.9119	0.9354	0.9750	1.0000	
Mg	0.655	0.6591	2.3126	0.9284	2.4928	0.9993	
Si	13.143	11.4404	1.4344	0.9377	1.5298	0.9999	
Ca	3.059	1.8657	0.9770	0.9542	1.0247	0.9991	
Zn	35.285	13.1966	1.2102	1.2129	0.9978	1.0000	
Al	0.475	0.4304	1.7897	0.9594	1.8692	0.9979	
C	0.000	0.0000	5.2279	0.9811	5.3287	1.0000	

Total 100.000 100.0000

Normalization factor = 2.1120

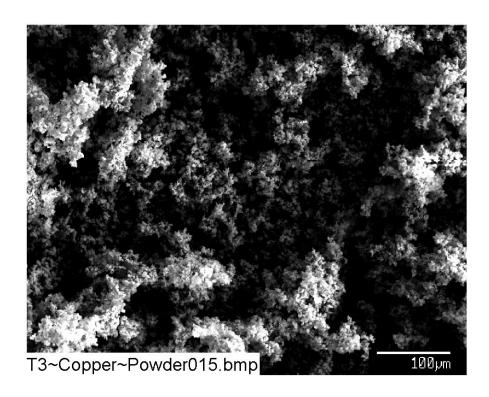


Figure D-6. SEM image magnified 500 times for a Test #3, Day-30 powder on a submerged copper coupon. (T3~Copper~Powder015)

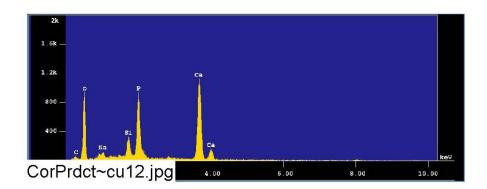


Figure D-7. EDS counting spectrum for the powder on copper shown in Figure D-6. (CorPrdct~cu12)

The results from the chemical composition analysis for CorPrdct~cu12 are given in Table D-3.

Table D-3. Chemical Compositions for CorPrdct~cu12, Figure D-7

#### May 9 2005

```
: NRC
Group
         : T3D30 ID# : 112
Sample
         : Corrosion product of Copper
Comment
Condition : Full Scale : 20KeV(10eV/ch,2Kch)
           Live Time : 60.000 sec
                                       Aperture #
                     : 15.0 KV
                                      Probe Current: 1.609E-09 A
           Acc. Volt
           Stage Point : X=17.909 Y=70.722 Z= 9.938
           Acq. Date : Mon May 9 15:34:51 2005
                                                 Net/Background
Element
           Mode
                   ROI (KeV)
                              K-ratio(%) +/-
                                         0.0036
                                                    6386 /
                                                               54
 OK
          Normal
                   0.25- 0.77
                              16.9583
Na K
          Normal
                   0.81- 1.27
                              0.3712
                                         0.0011
                                                    402 /
                                                               46
                              1.7009 0.0006
                                                    2479 /
                                                              254
 Si K
          Normal
                  1.50- 2.05
                                8.6109 0.0054
 PK
          Normal
                  1.75- 2.38
                                                   7744 /
                                                              164
                                                  13984 /
                               19.7450 0.0040
 Ca K
          Normal
                  3.39- 4.30
                                                               28
          Normal 0.09- 0.46
                              0.0000 0.0000
                                                      0 /
                                                              156
 CK
                           Chi square = 59.8005
                Atomic%
                          ZAF
                                  \mathbf{z}
Element Mass%
        51.054 70.0693 1.8119 0.9751 1.8582 1.0000
    0
         0.947
               0.9045 1.5353 1.0287 1.4923 1.0000
   Na
         3.260 2.5484 1.1533 0.9785 1.1852 0.9945
   Si
                8.6603 0.8538 1.1744 0.7286 0.9978
    P
        12.216
        32.523 17.8176 0.9913 0.9976 0.9936 1.0000
   Ca
```

0.000 0.0000 3.6900 1.0225 3.6090 0.9999

Total 100.000 100.0000 Normalization factor = 1.6616



Figure D-8. SEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl039)

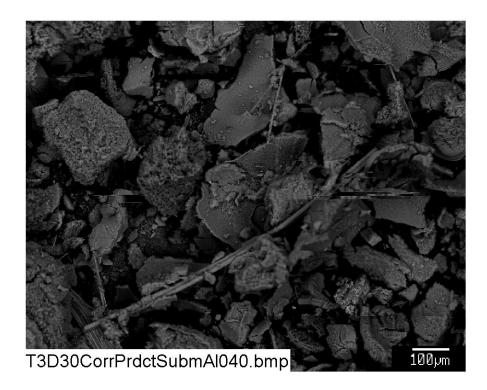


Figure D-9. Backscattered SEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdct SubmAl040)

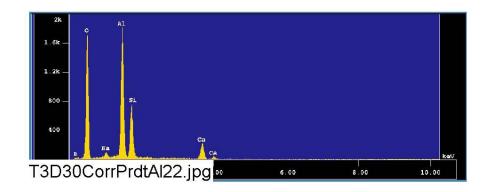


Figure D-10. EDS counting spectrum for the corrosion products (particles) shown in Figure D-9. (T3D30CorrPrdtAl22)

The results from the chemical composition analysis for T3D30CorrPrdtAl22 are given in Table D-4.

Table D-4. Chemical Compositions for T3D30CorrPrdtAl22, Figure D-10

May 17 2005

Group : NRC

Sample : T3D30 ID# : 25

Comment : Corr Prodct on Submerged Al

Condition: Full Scale: 20KeV(10eV/ch,2Kch)

Live Time : 60.000 sec Aperture # : 2
Acc. Volt : 15.0 KV Probe Current : 7.686E-09 A

Stage Point : X=17.100 Y=58.819 Z=11.000 Acq. Date : Tue May 17 16:07:30 2005

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Backgro	ound
ОК	Normal	0.25 - 0.77	43.5604	0.0044	10399 /	29
Na K	Normal	0.81- 1.27	0.7246	0.0010	497 /	45
Al K	Normal	1.26- 1.78	15.1592	0.0021	15016 /	184
Si K	Normal	1.50- 2.05	6.6963	0.0008	6185 /	674
Ca K	Normal	3.40- 4.30	4.2145	0.0082	1892 /	14
вк	Normal	0.00- 0.36	0.6943	0.0002	69 /	10

Chi\_square = 107.5732

```
Element Mass% Atomic% ZAF Z A F
O 52.728 58.8383 1.0170 0.9767 1.0413 1.0000
Na 1.120 0.8699 1.2989 1.0311 1.2612 0.9988
Al 19.542 12.9304 1.0831 0.9932 1.0934 0.9974
Si 10.480 6.6618 1.3149 0.9816 1.3397 0.9999
Ca 4.923 2.1928 0.9814 0.9916 0.9896 1.0001
B 11.207 18.5068 13.5610 1.1342 11.9568 1.0000
```

Total 100.000 100.0000

Normalization factor = 1.1902

Fe 2.399 1.5375 0.9896 1.0503 1.0060 0.9366

Total 100.000 100.0000

Normalization factor = 3.2486

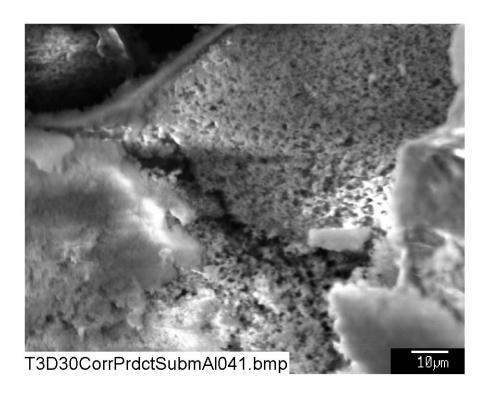


Figure D-11. SEM image magnified 1000 times for Test #3, Day-30 corrosion products on a submerged aluminum coupon. (T3D30CorrPrdctSubmAl041)

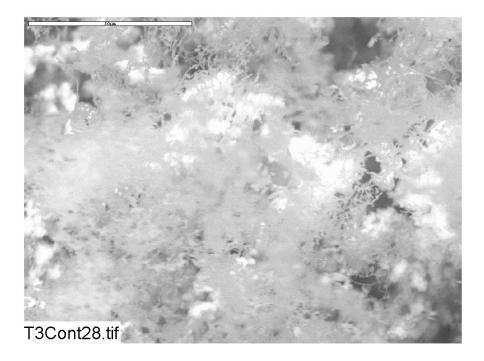


Figure D-12. ESEM image magnified 1000 times for Test #3, Day-30 corrosion products on a submerged concrete coupon. (T3cont28)

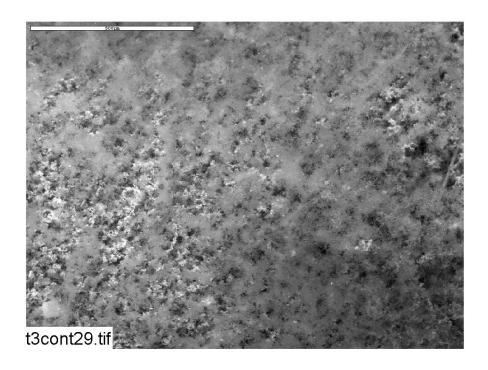


Figure D-13. ESEM image magnified 100 times for Test #3, Day-30 corrosion products on a submerged concrete coupon. (t3cont29)

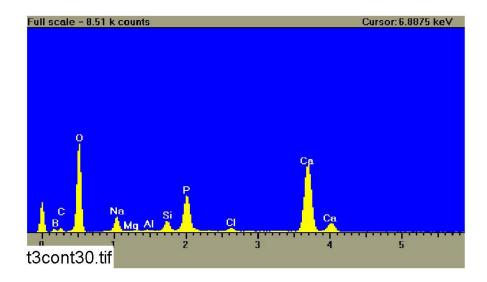


Figure D-14. EDS counting spectrum for the corrosion products (particles) shown in Figure D-13. (t3cont30)

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